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TITLE: THE USE OF INTERMEDIATE HEAT TREATMENTS FOR THE RESTORATION OF THE NOTCH-RUPTURE PROPERTIES OF INCONEL 718

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ABSTRACT

Inconel 718 is a widely used nickel-base alloy in the gas turbine engine industry. Many land based commercial/industrial power generating gas turbines as well as aircraft gas turbines contain critical components fabricated from this material. The effects of solution and intermediate heat treatments on the notch stress-rupture properties and microstructure of Inconel 718 were investigated. It was determined that a solution heat treatment of 1038°C for as short a time as 20 minutes could render the alloy notch sensitive by solutioning extensive quantities of the orthorhombic delta phase, Ni_3Cb. Restoration of notch ductility was accomplished by the insertion of a 917°C (10 h) intermediate heat treatment, prior to aging, during which significant quantities of the delta phase were precipitated. This intermediate heat treatment was not successful in restoring notch ductility to specimens which had been given longer, one hour, solution heat treatments at 1038°C. Various other intermediate heat treatments were also investigated for their effects on notch-rupture properties. It was determined that the most important factors controlling the notch-rupture ductility in Inconel 718 are the size, quantity and distribution of the delta phase. The grain size of the material did not directly control the notch properties.

INTRODUCTION

Today's marine/industrial as well as aircraft gas-turbines contain components fabricated from Inconel 718. This material is primarily used in its wrought forms but cast 718 material has also been used. This paper, however, will concern itself with wrought Inconel 718.

One of the more outstanding properties of this material is its weldability in the solution heat treated or aged condition. This is a result of the sluggishness of the precipitation hardening reaction in this alloy. The major hardening precipitate in Inconel 718 has been identified as a gamma" phase, body centered tetragonal, Ni_3 (Cb, Ti, Al).[1] Face-centered cubic gamma', consisting of the same constituents, exists as well and does contribute somewhat to strengthening. These precipitates are, therefore, different in composition than the conventional Ni_3 (Al, Ti) found in most nickel-base superalloys, and thus reflect the 5% columbium addition to this alloy. This difference in composition also manifests itself in the so-called overaging precipitate, the eta phase, which in 718 is orthorhombic, Ni_3 Cb, as opposed to hexagonal close-packed, Ni_3 Ti, which is normally found in other nickel-base alloys. Because of this, the eta phase in Inconel 718 is sometimes identified as delta phase in order to make the distinction and it will be referred to as such throughout this paper.

As superior as its weldability is, Inconel 718 is relatively difficult to form and to machine. Some forming operations that are easily performed with Inconel X-750, result in a high breakage rate for 718. Inconel 718 material has been conventionally solution annealed at 950°C-980°C, which does not always fully recrystallize the alloy. The more severe forming operations sometime require the material to be annealed at approximately 1035-1050°C. Material solution heat treated in this higher temperature range, possesses increased formability, but on subsequent aging develops into a notch sensitive condition in stress-rupture.

A recent investigation[2] was completed which detailed the notch-rupture properties of a specific heat of material which, in the context of this paper, shall be referred to as heat no. 1. Additional data from another heat of material (heat no. 2) will also be presented.

EXPERIMENTAL PROCEDURE

Commercial quality barstock was used in this investigation. Heat no. 1 was of rectangular cross-section, 50.8 mm by 76.2 mm; heat no. 2 was 63.5-mm-diam round stock. Table I lists the chemical composition for both heats. Both had been rolled from materials which had been vacuum induction melted and consumable vacuum remelted. Following rolling, the heat no. 1

TABLE I

CHEMICAL COMPOSITION OF HEAT NOS. 1 AND 2 IN WEIGHT %

	Heat No. 1	Heat No. 2
C	0.057	0.038
S	0.003	0.005
Mn	0.09	0.11
Si	0.15	0.18
Cr	18.80	18.85
Mo	3.07	3.09
Co	0.22	0.13
Ti	0.90	0.92
Al	0.49	0.49
B	0.005	0.004
Fe	Bal	Bal
Cu	0.03	0.05
Ni	52.35	52.25
P	0.006	0.006
Cb	5.12	5.02

barstock had been reportedly annealed at 982°C for 1 h and air cooled; heat no. 2 received a similar anneal, but at 954°C.

Both bars were machined into cylindrical notched and smooth bar specimens with gage lengths of 29.2 mm. The notch diameter and gage diameter of the notch specimens were nominally 4.53 mm and 6.4 mm respectively; the K_t of these specimens was approximately 3.5. The nominal gage diameter of the smooth bar specimens was 4.51 mm. Following machining, all of the specimens were given a solution heat treatment in argon for various times at predetermined temperatures. Subsequent to this initial heat treatment, some specimens received an intermediate heat treatment in argon and all were aged in argon as follows: 718°C (8 h), furnace cool at 55°C/hr to 621°C, 621°C (8 h) air cool.

Stress-rupture testing of the notched bars was conducted at 649°C and 690 MN/m^2. Smooth bar tests were conducted under the same conditions on selectively heat treated specimens, for comparative purposes.

Microstructural evaluation specimens were run in the furnaces at the same time as the actual test specimens. Standard preparation was used for optical microscopy and all samples were swab etched with a reagent consisting of one part 10 pct chromic acid and three parts hydrochloric acid.

For electron metallographic studies, the samples were electropolished in a solution of six parts methanol + one part sulphuric acid and one-half part nitric acid subsequent to 1μ diamond compound polishing. The samples were then electrolytically etched in the same solution and replicated.

RESULTS

Heat No. 1

Notch-Rupture Properties

For heat no. 1, essentially what was found was that the notch ductility was drastically reduced by as short a time as 20 min. at 1038°C, when compared to conventionally solution heat treated, 954°C (1 h), material (Table II). The data also show that an initial attempt at restoring notch ductility utilizing a 954°C (3 h) intermediate heat treatment was unsuccessful; the 917°C (10 h) intermediate heat treatment was successful in restoring notch ductility (life), but only for the specimens exposed to the shorter, 20 min., solution heat treatment. This same intermediate heat treatment was unsuccessful in restoring notch life to material solution heat treated at 1038°C for 1 hour. Smooth bar rupture properties for heat no. 1 are given in Table III for comparative purposes.

TABLE II
HEAT NO. 1

RESULTS OF NOTCH-RUPTURE TESTING AT 690 MN/m^2, 649°C*

Specimen Number	Solution Heat Treatment	Intermediate Heat Treatment	Time To Rupture Hrs.
1	1038°C (20 min)	None	0.4
2	" "	"	0.3
3	" "	"	0.3
4	1038°C (1 hr)	"	0.1
5	" "	"	0.2
6	954°C (1 hr)	"	153.5
7	" "	"	423.1**
8	917°C (10 hrs)	"	424.1
9	" "	"	143.2
10	1038°C (20 min)	954°C (3 hrs)	0.5
11	" "	" "	0.4
12	1038°C (20 min)	917°C (10 hrs)	882.8**
13	" "	" "	499.5**
14	" "	" "	838.4**
15	" "	" "	838.4**
16	1038°C (1 hr)	954°C (3 hrs)	0.3
17	" "	" "	0.6
18	1038°C (1 hr)	917°C (10 hrs)	0.5
19	"	" "	0.8
20	" "	" "	1.1

* All specimens given an aging treatment of 718°C (8 hrs), furnace cool at 55°C/hr to 621°C, 621°C (8 hrs), air cool following solution or solution and intermediate heat treatments.

** Test discontinued.

TABLE III

HEAT NO. 1

RESULTS OF SMOOTH BAR RUPTURE TESTING AT 690 MN/m^2, 649°C*

Specimen Number	Solution Heat Treatment	Intermediate Heat Treatment	Time to Rupture Hrs.	Percent Elong.
1	1038°C (20 min)	None	79.9	4.1
2	" "	"	71.0	4.3
3	1038°C (1 hr)	"	88.0	4.2
4	" "	"	112.4	4.9
5	954°C (1 hr)	"	48.9	5.3
6	" "	"	58.7	5.4
7	917°C (10 hrs)	"	23.7	9.7
8	" "	"	33.0	10.1
9	1038°C (1 hr)	954°C (3 hrs)	42.0	4.1
10	" "	" "	35.9	4.1
11	1038°C (1 hr)	917°C (10 hrs)	44.4	7.4
12	" "	" "	56.8	8.2

* All specimens given an aging treatment of 718°C (8 hrs), furnace cool at 55°C/hr to 621°C, 621°C (8 hrs), air cool following solution or solution and intermediate heat treatments.

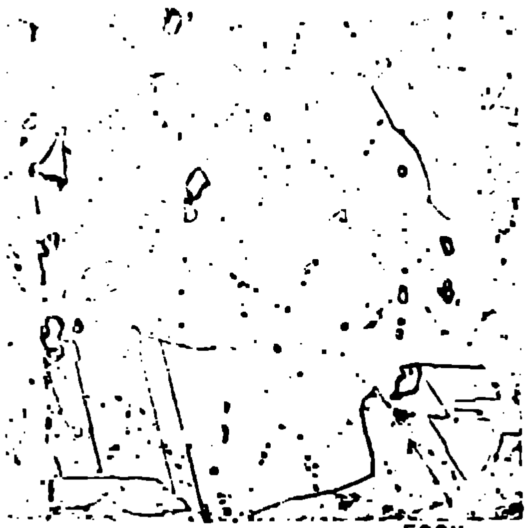
Microstructure

The grain size of the heat no. 1 barstock was ASTM 6-8, as received. Solution heat treatments at 1038°C increased the grain size to ASTM 4-7 and ASTM 3-5, respectively, after a 20 min. or 1 hour duration at temperature (Figure 1). After 20 min. at temperature, all that remained of the delta phase was in the form of a fine prior grain boundary precipitate. The one-hour solution heat treated material was completely devoid of delta phase. These observations, therefore, show that grain growth can occur before complete dissolution of the delta phase.

The fact that the 917°C (10 h) intermediate heat treatment restored notch ductility, however, indicates that increased grain size of ASTM 4-7 was not the cause of the loss of notch ductility; rather this was apparently caused by a lack of an adequate distribution of properly sized delta phase plates in the microstructure. This intermediate heat treatment produced greatly increased quantities of delta phase over that residual amount present after the 20 min. 1038°C solution heat treatment.

Fig. 2 shows that the delta phase produced by this specific intermediate heat treatment was of the same general size and distribution as that of conventionally solution heat treated, 954°C (1h) material, which was also notch ductile. It should be pointed out that the lower temperatures employed in the study (917°C and 954°C) did not alter the grain size when used as either solution or intermediate heat treatment temperatures. The top group of photomicrographs in Figure 2 was taken of the material which received a 1038°C (20 min.) solution heat treatment followed by a 954°C (3 h) intermediate heat treatment prior to aging. These show a noticeably smaller quantity of delta phase than present in the other groups of photomicrographs in this figure. When examined carefully, particularly with respect to the election photomicrographs, the top group in Fig. 2 appears to display a generally smaller delta phase plate size (perhaps only half as large on the average) than those present in the others.

Fig. 3 shows the microstructure of the material solution heat treated for one hour at 1038°C followed by a 917°C (10 h) intermediate heat treatment. The extensive precipitation of the delta phase and the large size of these plates, compared to the photomicrographs in Fig. 2 should be noted. There was also a noticeably substantial reduction of intragranular delta phase upon aging. This was not evident in any of the other microstructure, except perhaps to a very slight degree for that material which received 1038°C (20 min.) + 917°C (10 h) + age. Despite the noticeable reduction in the amount of delta phase for the 1038°C (1 h) + 917°C (10 h) + aged material, substantial quantities of relatively large size delta plates remained in the structure. It was not the intent of this investigation to dwell on the reason for the reduction in delta phase content upon aging but it appears to be related to equilibrium conditions during aging with respect to competition for Cb from the gamma"/gamma' precipitates. The interesting point is that, in this case, the aged microstructure seemingly displayed an adequate amount of delta phase but was still not restored to



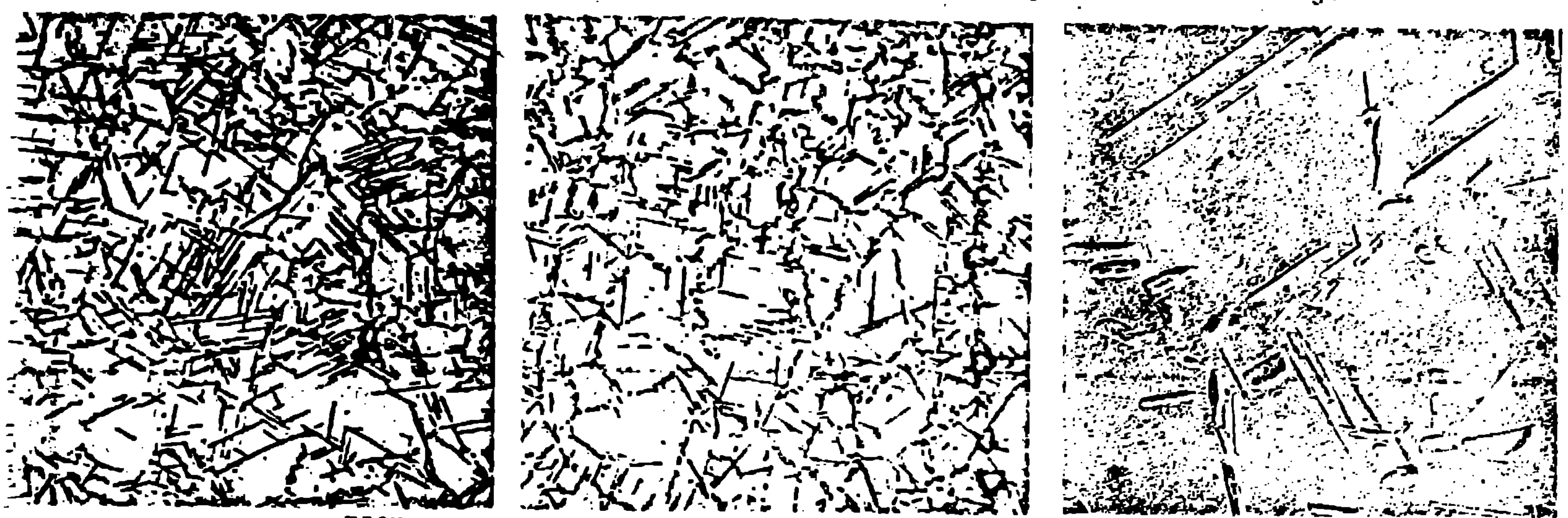
1038°C (20 min)

1038°C (1 hr)

Fig. 1

PHOTOMICROGRAPHS SHOWING PRESENCE OF PRIOR GRAIN BOUNDARY PRECIPITATE IN SPECIMEN RECEIVING SHORTER SOLUTION HEAT TREATMENT AT 1038°C.

1038°C (20 min) + 954°C (3 hrs) Left: SHT + IHT, Center & Right: SHT + IHT + Age

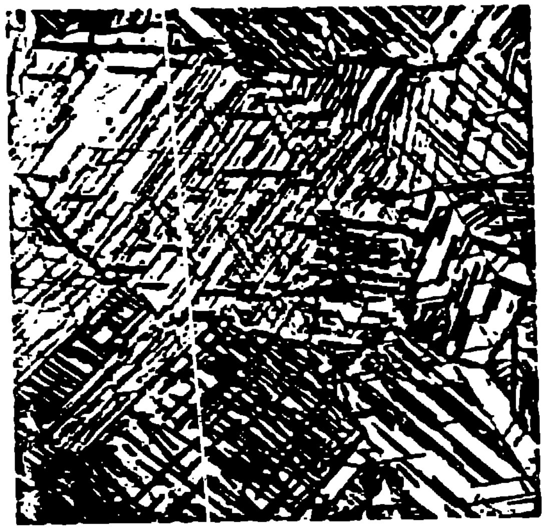


1038°C (20 min) + 917°C (10 hrs) Left: SHT + IHT, Center & Right: SHT + IHT + Age

954°C (1 hr) Left: SHT, Center & Right: SHT + Age

FIGURE 2

MICROSTRUCTURES SHOWING δ PHASE AMOUNT, DISTRIBUTION AND PLATE SIZE DIFFERENCES OF SEVERAL DIFFERENTLY HEAT TREATED SPECIMENS.



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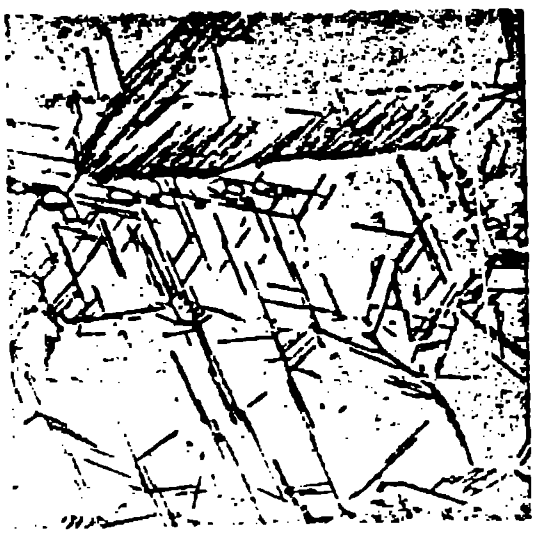
1038°C (1 hr) + 917°C (10 hrs)



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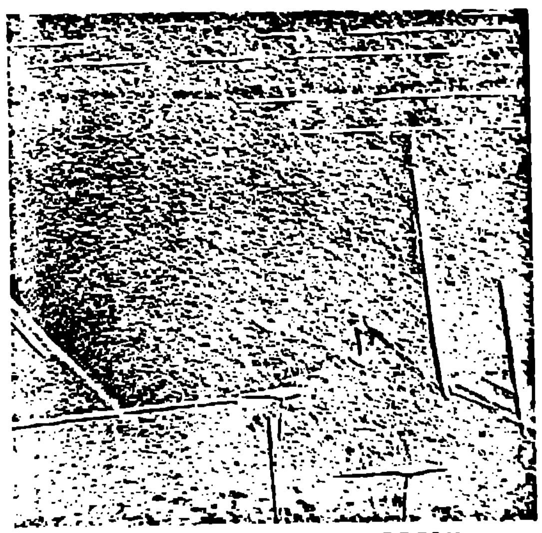

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1038°C (1 hr) + 917°C (10 hrs) + Age

Fig. 3

MICROSTRUCTURES SHOWING A LOWERING IN THE AMOUNT OF δ PHASE ON AGING, AND THE δ PHASE PLATE SIZE TYPICAL OF SPECIMEN RECEIVING THIS COMBINATION OF SHT AND IHT.

a notch ductile condition. Furthermore, the delta phase plate size was larger (on the average almost twice as large) than that which existed for the notch ductile groups of specimens, one of which had received the same intermediate heat treatment. Therefore, it can be said that the length of time of the solution heat treatment, as manifested as increased grain growth (ASTM 3-5 versus 4-7) resulted in a different delta phase plate size as well, upon subsequent heat treating.

Heat No. 2

Notch-Rupture Properties

Table IV shows the notch-rupture data for heat no. 2. Here again 1038°C, for as short a time as 20 min., produced a notch-rupture problem. It is also obvious that a longer time solution heat treatment of 1038°C for 1-1/2 hours produced a material which was not capable of being restored to a notch ductile condition. The most interesting and important result of the testing of heat no. 2 is the fact that notch ductility was restored to the material having received a solution heat treatment of 1038°C (20 min.) by a 954°C intermediate heat treatment for only one hour. It should be recalled that we were unable to restore notch life to heat no. 1 material using a 954°C (3 h) intermediate heat treatment. Table V shows the smooth bar rupture properties for heat no. 2.

Comparison Microstructures

Fig. 4 shows the photomicrographs of the as received material from heat no. 2 along with that from heat no. 1. Heat no. 1 had a somewhat coarser grain size (ASTM 6-8) than did heat no. 2 (ASTM 9). Furthermore, heat no. 1 appeared to display considerably smaller delta phase plates as received than did heat no. 2 and perhaps even somewhat fewer plates. Fig. 5 shows the respective microstructure after receiving a 1038°C (20 min.) + 954°C (3 h) + age heat treatment. Note the larger increase in grain size for heat no. 1 than for heat no. 2 (ASTM 4-7 vs ASTM 8), caused by the initial 20 min. duration at the 1038°C temperature. It is also observed that the amount of delta phase was more plentiful for heat no. 2. This was particularly true with respect to the amount of intergranular delta phase.

DISCUSSION

From the work described, it appears that the ease of using a ductility restoration heat treatment is directly dependent upon the starting grain size and delta phase distribution. Heat no. 1 had a somewhat coarser grain size but probably more importantly, smaller and also perhaps fewer delta phase plates in grain boundaries than did heat no. 2. Upon high temperature solution heat treating, the grain growth resistance of heat no. 2 was superior to that of heat no. 1. Consequently, when subsequent intermediate heat treatments were applied, heat no. 2, with more sites (grain boundaries) for delta precipitation, was restored to a notch ductile condition in an easier fashion (at a temperature further from the nose of the delta precipitation curve,[3,4] and in a shorter time).

TABLE IV

HEAT NO. 2

RESULTS OF NOTCH-RUPTURE TESTING AT 690 MN/m^2, 649°C*

Specimen Number	Solution Heat Treatment	Intermediate Heat Treatment	Time to Rupture Hrs.
1	1038°C (20 min)	None	0.6
2	" "	"	0.4
3	" "	"	600.3
4	" "	"	0.6
5	" "	"	2.0
6	1038°C (1h)	None	0.7
7	" "	"	0.5
8	954°C (1h)	None	409.3 **
9	" "	"	605.8
10	" "	"	668.2 **
11	" "	"	674.9 **
12	1038°C (20 min)	954°C (1h)	419.2 **
13	" "	" "	699.1 **
14	" "	" "	666.8 **
15	" "	" "	685.3 **
16	1038°C (20 min)	954°C (3h)	417.6 **
17	" "	" "	683.8 **
18	" "	" "	685.4 **
19	" "	" "	685.4 **
20	1038°C (1-1/2h)	954°C (1h)	0.3
21	" "	" "	0.5
22	1038°C (1-1/2h)	954°C (3h)	0.4
23	" "	" "	0.1
24	1038°C (1-1/2h)	954°C (6h)	0.3
25	" "	" "	1.0

* All specimens given an aging treatment of 718°C (8h), furnace cool at 55°C/hr to 621°C, 621°C (8h), air cool following solution or solution and intermediate heat treatments.

** Test discontinued.

TABLE V

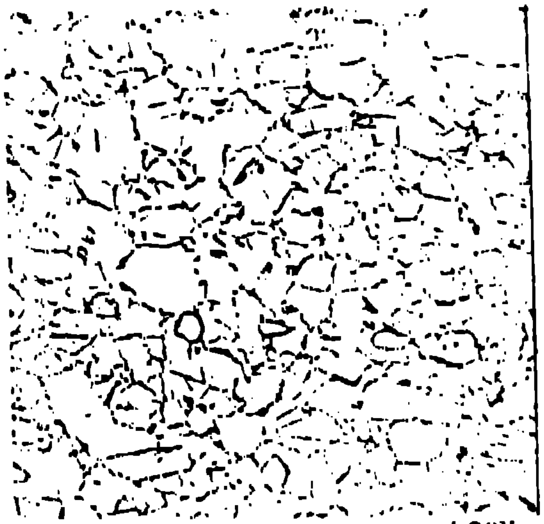
HEAT NO. 2

RESULTS OF SMOOTH BAR RUPTURE TESTING AT 690 MN/m^2, 649°C*

Specimen Number	Solution Heat Treatment	Intermediate Heat Treatment	Time to Rupture (hrs)	Percent Elong.
1	1038°C (20 min)	None	299.2	10.8
2	" "	"	351.0	6.9
3	" "	"	208.4	7.2
4	" "	"	100.3	6.4
5	954°C (1h)	None	72.3	10.0
6	" "	"	146.2	11.3
7	" "	"	16.9	16.7
8	1038°C (20 min)	954°C (1h)	122.9	9.0
9	" "	" "	27.1	9.0
10	" "	" "	97.0	15.0
11	" "	" "	194.5	11.9
12	1038°C (20 min)	954°C (3h)	93.0	13.8
13	" "	" "	39.4	9.7
14	" "	" "	90.7	16.4
15	" "	" "	111.4	10.0

* All specimens given an aging treatment of 718°C (8h), furnace cool at 55°C/hr to 621°C, 621°C (8h), air cool following solution or solution and intermediate heat treatments.

Heat No. 1



Heat No. 2

Fig. 4

PHOTOMICROGRAPHS OF MATERIAL AS RECEIVED FROM BOTH HEATS.

500X

Heat No. 1

500X

Heat No. 2

Fig. 5

MICROSTRUCTURES OF SAMPLES FROM BOTH HEATS WHICH RECEIVED $1038^{\circ}C$ (20 MIN) + $954^{\circ}C$ (3 HRS) + AGE.

The dependence of notch life (ductility) on the delta phase precipitation characteristics including the size of the plates has also been shown previously in several investigations.[5,6,7] One might postulate that a specific grain size might almost be predisposed to influence the precipitation of delta phase plates of a size and morphology that is inconsistent with that grain size for notch ductility considerations; this might be the case for the 1038°C (1 h) + 917°C (10 h) heat treated material from heat no. 1.

The grain size-delta phase interrelationship seems to basically be one in which delta phase can pin grain boundaries and , therefore, inhibit grain growth. Once the delta phase is completely or nearly completely dissolved, grain growth can proceed, as one might expect. As grain boundaries themselves act as preferred sites for subsequent formation of delta phase during an intermediate anneal, it is important to initially produce a relatively fine grained material containing substantial quantities of intergranular delta phase through proper control of thermomechanical processing and annealing procedures by the mill.

CONCLUSIONS AND RECOMMENDATIONS

Thus far, of course, we have not demonstrated that we can restore notch ductility to material solution heat treated for longer than 20 minutes at 1038°C. This problem is compounded by the fact that, as has been shown, different heats of material require different restorative intermediate heat treatments for even the shorter time solution heat treatments. Obviously it would be too time consuming and expensive to make a separate determination for each and every heat of material that one might possibly be using for a specific application requiring good formability during processing and acceptable notch-rupture properties in service.

A possible solution to this problem is simply more careful control of thermomechanical processing. This could be accomplished by instituting a special specification such as within the AMS system which would state appropriate grain size and delta phase distribution and size requirements that are proven to permit notch ductility restoration by a specifically determined intermediate heat treatment. Naturally a good deal of applied development would be needed to determine the actual specification limits. This type of more stringent specification might be likened to that for the low and extra-low carbon versions of 304 stainless steel or to the extra low interstitial specifications for some titanium-base alloys.

The fabricating mills should not incur any additional costs due to such a specification in that their normal quality control and sampling procedures would allow them to direct the appropriately processed (i.e., grain size, delta phase distribution) material so as to be identified according to that more stringent specification.

It should be pointed out that only limited quantities of the higher quality Inconel 718 would probably be needed. However, it would be a help to manufacturing metallurgists and production engineers. It would allow them to specify the higher quality material for those situations where the fabrication of a creep-rupture limited part is difficult, thereby requiring the higher temperature annealing procedures in process, and restoration procedures during final heat treatment.

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